

INTERLABORATORY
STUDY 94-1
NOVEMBER 1994

**PART A: TRACE METALS SPIKED INTO
PRECIPITATION
PART B: TRACE METALS SPIKED ONTO
FILTERS**

IN SUPPORT OF

THE INTEGRATED
ATMOSPHERIC
DEPOSITION
NETWORK
(IADN)

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1 SUMMARY OF INTERLABORATORY STUDY 94-1

Interlaboratory Study 94-1 was the third study for Trace Metals analysis initiated in support of the Integrated Atmospheric Deposition Network (IADN) to provide an assessment of between-laboratory variability. Participation was limited to laboratories which contribute to the IADN database or related programs. This study was sponsored by the Canada-Ontario Agreement (COA) Air Toxics Workgroup, and conducted as a joint project between the Atmospheric Environment Service (AES) of Environment Canada and the Quality Management Unit (QMU), Laboratory Services Branch (LSB) of the Ontario Ministry of Environment and Energy (MOEE).

The study was divided into two parts: Part A consisting of Trace Metals spiked into a bulk precipitation sample, and Part B consisting of a Trace Metals solution being spiked onto filters used for ambient air sampling. Six laboratories participated in Part A and four laboratories participated in Part B. The parameter list consisted of 8 different elements. Results were received from all participating laboratories.

The overall interlaboratory performance in Part A (precipitation) of this study has shown improvements (Aluminum and Chromium) or a maintenance of performance from the previous studies among these laboratories^{4,5}, except for Selenium. Each laboratory had individual problems with different Trace Metals. Comparison with reference materials should help improve some of these problems. The low target levels for many of these Trace Metals were beyond the analytical capability of several of the participants. As the IADN network is a research program, the lowest possible levels of detection are to be desired from the laboratories providing data. Further method development to improve detection capabilities and to improve between-laboratory variability at these low concentrations is recommended.

The analysis of the spiked filters in Part B was the first time the interlaboratory performance had been compared among these laboratories. While there were individual problems with some of the parameters, the differences among the laboratories were not extreme. Future intercomparisons should help improve the comparability among these laboratories.

2 INTRODUCTION

Interlaboratory performance studies are conducted to assess the comparability and accuracy of data among different laboratories. These studies are useful for the identification of biases, precision and accuracy problems, as well as ensuring data quality. Participation in such studies can serve as a guide for improving individual laboratory performance and maintaining performance standards.

This study was the third in a series designed to assess the analytical variability among laboratories contributing to the Integrated Atmospheric Deposition Network (IADN). IADN was established as a joint venture between Canada and the United States under the direction of the International Joint Commission¹. The intent of IADN is to identify toxic airborne substances in the Great Lakes Basin, and by means of the network, quantify the total and net atmospheric loadings of these contaminants, and define spatial and temporal trends in the atmospheric deposition of these substances. Data from several participating agencies is to be merged into a central database. Comparability of these contributing data sets is an important component of the IADN Quality Assurance Implementation Plan². This interlaboratory study provides information to help establish the comparability of data sets and is a recommended activity of the IADN Quality Assurance Program Plan³. Sponsorship of this interlaboratory study was through the Canada-Ontario Agreement (COA) Air Toxics Workgroup. Co-ordination of the study and preparation of all materials was done by the Quality Management Unit (QMU) of Laboratory Services Branch (LSB) of the Ontario Ministry of Environment and Energy (MOEE).

Interlaboratory Study 94-1 targets laboratories analyzing for Trace Metals in ambient air and/or precipitation. The aim of this study was to evaluate the comparability of the analysis of spiked matrix samples among the participating laboratories. The study was divided into two parts: Part A consisting of Trace Metals spiked into a bulk precipitation sample, and Part B consisting of a Trace Metals solution being spiked onto filters used for air sampling. The first study in this series was conducted in 1992⁴ and the second in 1993⁵.

A list of participants is given in Appendix 2. Each participant was assigned a unique identification code for ease in data manipulation.

Section 3 describes sample preparation, sample distribution, analytical methodology, and data evaluation procedures. Final results are tabled in Appendix 1 and discussed in Section 4.

3 PROCEDURE

3.1 Preparation of Spiking Solutions

The QMU of LSB, MOEE provided individual concentrated stock solutions of the eight Trace Metals to be used in this study. These solutions had been previously verified against US-EPA materials and used extensively for LSB in-house Performance Evaluation samples and MOEE interlaboratory studies. Two separate spiking solutions were prepared by diluting appropriate aliquots of the concentrated stock solutions in distilled, deionized water (DDW). Concentration levels were designed to approximate similar levels of the Trace Metals found in precipitation and ambient air. The solutions were preserved with 2% concentrated nitric acid. The concentration levels were confirmed by ICP-Mass Spectrometry (ICP-MS).

3.2 Part A: Preparation of Spiked Precipitation

A bulk precipitation sample was prepared by collecting the remainder of routine precipitation samples received for analysis at LSB. The bulk sample was filtered after collection using a Whatman 2V Qualitative filter to remove algae, and stored in a Nalgene container at 4°C in the dark. The concentration level of the Trace Metals of interest was checked using ICP-MS. These levels were high for some of the Trace Metals, compared to the designed spiking concentrations, so the bulk sample was diluted 50:50 with DDW to become the matrix for the study.

Prior to preparing the spiked samples, the matrix (the diluted precipitation) was stirred for an hour. The matrix was dispensed into three 2-litre volumetric flasks. Flask one was filled to the mark and designated as the "blank". Flask two was designated the low spike, spiked with the appropriate aliquot of the spiking solution, and filled to the mark. Flask three was designated the high spike and treated in a similar manner as flask two. All three solutions were well mixed before dispensing into the individual sample bottles.

As part of the invitation to participate in this study, laboratories were asked whether they received their samples preserved or not. As some participants required unpreserved samples, the three solutions were not preserved with nitric acid.

The letter of invitation also indicated that the samples would be shipped in polyethylene (PET) bottles. If these bottles were not suitable, participants were asked to supply a set of sample bottles of the type routinely used for the submission of IADN samples. The bulk samples were dispensed into the sample bottles, either PET or those provided by individual participants. Two sets of bottles were supplied pre-charged with nitric acid. These have been indicated in Table 3, Appendix 1. The "blank" sample was labelled "94-1-MET1", the low spike was labelled "94-1-MET2", and the high spike was labelled "94-1-MET3". All subsequent reference to these samples omits the prefix "94-1-".

3.3 Part B: Preparation of Spiked Filters

All participants were requested to supply a minimum of three filters of the type they routinely used for ambient air sampling for the IADN program. They were also asked to indicate if all or only part of the filter was actually analyzed. For those participants who analyzed only part of the filter, the appropriate size was cut from the filters provided. All filters were handled with forceps.

One filter (or strip) was designated the "blank". It was left exposed to the laboratory environment for the same length of time as the spiked filters were exposed for drying. One participant requested two "blank" strips. The second filter was designated the low spike, and an appropriate aliquot of the spiking solution was carefully dispensed onto the filter. The filter was "hung to dry" in a fumehood, using a nylon line and plastic clothes pegs. The third filter was designated the high spike and treated in a similar manner as the second filter. When all the filters were dry, they were individually packaged into labelled plastic 'Zip-lok' bags or plastic holders. The "blank" sample was labelled "94-1-MET4", the low spike was labelled "94-1-MET5", and the high spike was labelled "94-1-MET6". All subsequent reference to these samples omits the prefix "94-1-".

3.4 Sample Distribution

The precipitation samples were packed into boxes and shipped by Purolator Courier to the participating laboratories. A list of the laboratories receiving sample sets is given in Appendix 2. Samples were shipped on November 21, 1994. A copy of all correspondence is also included in Appendix 2.

The packaged filters were packed either in lined envelopes with cardboard dividers or in a box for shipping by Purolator Courier to the participating laboratories. A list of the laboratories receiving sample sets is given in Appendix 2. Samples were shipped on November 29, 1994. A copy of all correspondence is also included in Appendix 2.

3.5 Analytical Methodology

Participating laboratories were requested to analyze the samples using their routine in-house methods used to analyze ambient air or precipitation samples for the IADN program. Participants receiving the unpreserved precipitation samples were advised that they could preserve the samples upon receipt, if that was their routine procedure, and asked to indicate this on the report form. Laboratories receiving the spiked filters were instructed to analyze the entire filter, as there is no guarantee of homogenous dispersion of the spiking solution on the filter. Participants were also requested on the report form provided (Appendix 2) to indicate the sample treatment prior to analysis, the Instrument used, and any dilution factors (if necessary). All participants were assigned a unique identification code.

3.6 Data Reporting

Results were submitted to the QMU, LSB in written form. All data were manually entered by laboratory code into an electronic spreadsheet. For Part A, all results were entered into the spreadsheet in units of $\mu\text{g/L}$. For Part B, all results were entered into the spreadsheet in units of $\mu\text{g/Filter}$. All participants submitted results for the samples they received.

The participating laboratories were mailed a copy of the tables of results (Tables 1A and 2A) from both Part A and B on April 10, 1995. Laboratory 9414 had submitted their results for sample MET4 all qualified with ">", which was entered into the table of results. They reported that the qualifier should be "<".

The results for the spiked samples (MET2, MET3, MET5, and MET6) were blank corrected (i.e. the results for MET1 were subtracted from MET2 and MET3. The results for MET4 were subtracted from MET5 and MET6). All blank-corrected values are included in Tables 1B and 2B, Appendix 1. For these calculations, results reported with a "<" or "ND" were treated as "0".

The interlaboratory mean, median, standard deviation (SD), and relative standard deviation (%RSD) were calculated for each element, both as reported and blank corrected, and are included in Tables 1A, 1B, 2A, and 2B, Appendix 1. Laboratories whose detection limit was greater than the target value for some of the elements, did not have their results included in these calculations. As the data set is small, these calculated values are provided as an approximate indicator of the spread of the data, and may not necessarily be statistically correct.

Table 3 lists the containers, preservative, preparation procedures and instruments used by the participants analyzing the precipitation samples. Table 4 lists the filter types, preparation procedures and instruments used by the participants analyzing the spiked filters.

4 DISCUSSION

4.1 Part A: Overview of Interlaboratory Performance

Results were received from all of the laboratories which received the precipitation samples.

The interlaboratory mean, median, and standard deviation for the reported (i.e. not blank corrected) MET2 and MET3 results (Table 1A) are included for information only. They do not give an accurate assessment of agreement with the study target values, as at least one participant detected each element in the unspiked matrix sample, MET1. This suggests that some of these elements are present in all of the precipitation samples, and therefore affects the total result being reported for MET2 and MET3. The following discussion is based on the blank-corrected values for MET2 and MET3 (Table 1B).

To aid in evaluating the results, a graphical technique was used. As each parameter had a pair of results at two concentration levels, these results may be plotted on an X-Y plot using the Youden technique⁶. The result from the "low" sample is plotted on the vertical axis and the result from the "high" sample is plotted on the horizontal axis. The blank-corrected values were used to prepare these graphs. The interlaboratory mean is also plotted on the graphs.

The graphs are divided into four quadrants, with the intersection point at the target values. The data points should cluster around the target if random error is the only source of variability. Results in the upper right quadrant are considered biased high and those in the lower left quadrant are biased low. The main source of this type of variability is a difference in analytical standards or inadequate calibration practices. Data points that fall in the lower right or upper left quadrants are considered erratic or out-of-control. Sources of this type of error are more difficult to ascertain. Sources of erratic performance may include contamination during sample preparation procedures, dirt in the instrument nebulizer causing poor aspiration (ICP or AAS) or scattered light emissions.

Within-laboratory precision may be assessed by drawing a line between the origin and the intersection of the target values. The closer the data point is to this diagonal line, the better the within-laboratory precision.

The results for Aluminum (Figure 1) indicate that the primary difference between the laboratories is a difference in standards. Only Laboratory 9412 had results that were not in the upper right or lower left quadrant (see individual discussion below). The %RSD for Aluminum is less than 19%, which is an improvement over the last study⁵ among these laboratories. Two of the ampouled standards in the previous study were at the same concentration level as the two samples in this study. As only one participant performed any pre-treatment on the samples prior to instrumental analysis (Table 3), the results from the two studies can be directly compared for all of the Trace Metals. The %RSD for IADN1 (target = 25 µg/L) and IADN3 (target = 100 µg/L) in Study 93-1 were 54% and 52%, respectively. As

standards should have no interferences and therefore be "easier" to analyze, it would be expected that interlaboratory performance would have been better in the previous study⁵ than in this study. The improvement in between-laboratory performance in this study is encouraging, though as a group there is a slight low bias. Ongoing comparison of their standards to international reference standards needs to be maintained.

The target level for Arsenic was below the detection limit of two of the participants (9411 and 9414). The results (Figure 2) from the other participants are divided into two groups, Laboratories 9412 and 9415, and Laboratories 9416 and 9413. The former pair are biased high and the latter pair have good agreement with the target. The differences between the laboratories appears primarily due to a difference in standards and may be resolved through comparisons with reference standards. The target levels in this study are considerably lower than those in the previous studies^{4,5}. However the between-laboratory variability in this study (%RSD 11-12% for the corrected values) compares well with the previous studies (2.5-8% in Study 93-1 and 7-18% in Study 92-4).

The target level for Cadmium in MET2 was below the detection limit for two of the participants (9412 and 9413), but the target level for MET3 was within their analytical range. Their results have been plotted on the graph (Figure 3), but are marked with brackets, as their performance can only be evaluated for MET3. The results demonstrate considerable between-laboratory variability. The %RSD is high in this study (55-58%, Table 1B), compared to the previous two studies^{4,5}, which had %RSD in the range 5-18%. However the target levels in this study are lower than in the previous two studies, and this may have contributed to the increased between-laboratory variability. In both of the previous studies, the highest %RSD was observed for the lowest concentration samples.

The Chromium results demonstrate good agreement among the participants and with the target, for all but Laboratory 9411 (Figure 4). See below for discussion of Laboratory 9411's results. The between-laboratory variability has improved in this study, despite the low target levels. The %RSD in this study is 10-17%, while in Study 93-1 the %RSD range from 8-76%⁵ and in Study 92-4⁴ the %RSD ranged from 5-35%. The improvement in between-laboratory performance is particularly notable at the 0.5 µg/L level (10% for sample MET2 in this study compared to 76% for sample IADN1 in Study 93-1⁵).

The target level for Copper in MET2 was below the detection limit for Laboratory 9414, but the target level for MET3 was within their analytical range. Their results have been plotted on the graph (Figure 5), but are marked with brackets, as their performance can only be evaluated for MET3. The results demonstrate agreement among three of the participants, but all of the results are biased high relative to the target. The previous studies^{4,5} found that the participants had an intercept bias for Copper of 1.0 - 5.0 µg/L. This would affect the results at the low target levels of this study, contributing to the overall high bias among the participants. In Study 93-1 the interlaboratory mean for IADN1 was more than double the target value (1.049 µg/L). In this study, the interlaboratory mean for MET2 was 60% higher than the target (0.52 µg/L) and for MET3 the interlaboratory mean was 25% higher than the target (2.10 µg/L). These results indicate an improvement in the intercept bias among the participants, but further efforts are required to reduce this bias.

The Lead results (Figure 6) demonstrate considerable between-laboratory variability. As with Cadmium, the target levels in this study are lower than the

previous two studies^{4,5}, possibly contributing to the high %RSD (23-47%) in this study. In Study 93-1⁵, sample IADN1 had a target level of 2.49 and the %RSD for that sample was 35%, which is similar to the performance in this study. In the previous two studies, the %RSD decreased as the sample concentration increased.

Only three participants reported results for Selenium, and of these, only two were able to analyze for Selenium at the target level. Figure 7 demonstrates that the two participants who could analyze Selenium at this low level do not agree with each other or the target. This was poorer performance than the previous two studies^{4,5}. Comparison with international reference standards is a priority for this element.

The Zinc results form two groups among the participants, Laboratories 9412 and 9414, and Laboratories 9411, 9413, and 9416 (Figure 8). The latter group demonstrate good within-laboratory precision, as all data points are along the diagonal line. The former group appear erratic or out of control. Since they are biased high for the low sample (MET2) and biased low for the high sample (MET3), it appears that Laboratories 9412 and 9414 may have a high intercept and possibly a low slope for their calibration curve. The intercept problem was identified in the previous two studies^{4,5}, and may still be a problem for these two laboratories.

As the analytical procedure for all but one of the participants was the same in Part A of this study as the previous studies^{4,5} (i.e. direct instrumental analysis of the solutions), a direct comparison of performance can be made. The overall performance of this study has shown improvements (Aluminum and Chromium) or a maintenance of performance from the previous studies^{4,5}, except for Selenium. The low target levels for many of these Trace Metals were beyond the analytical capability of several of the participants. These levels were based on annual averages for precipitation as reported by the National Water Research Institute⁷. As the IADN network is a research program, the lowest possible levels of detection are to be desired from the laboratories providing data. Further method development to improve detection capabilities and to improve between-laboratory variability at these low concentrations is recommended.

4.2 Part A: Individual Laboratory Performance

The participants in this study are the same laboratories that participated in Interlaboratory Study 92-4⁴ and/or Interlaboratory Study 93-1⁵. To preserve confidentiality, the laboratories have been assigned different ID Codes in this study. Therefore the following individual laboratory review is based only on the results of this study, with no comparisons to the previous studies. Unless indicated, the review of performance is based on the blank corrected results for MET2 and MET3.

Laboratory 9411

Laboratory 9411 used a 4X dilution factor to obtain results for Zinc in MET2 and MET3, and Aluminum in MET3. The target levels for Arsenic were below their detection limit.

Laboratory 9411 was biased high for all the Trace Metals, except for Chromium in MET2 and Zinc in both samples. The use of a dilution factor for Zinc, as noted

above, may have introduced the low bias. Their performance for Chromium was erratic (Figure 4), with their result for MET2 agreeing with the target, while they were biased high for MET3. This suggests a possible slope problem with their calibration curve. As no preparation steps were used on the samples (other than the dilution as noted above), the source of the high bias most likely is due to their calibration standards. Comparisons with international reference standards should help resolve this problem.

Laboratory 9412

Laboratory 9412 noted that for their results, "N/A" indicated Not available, sample consumed before valid results were obtained. They also used a 5X dilution to obtain results for Aluminum, Copper, Lead and Zinc in sample MET3. The target level for Cadmium in MET2 was below their detection limit.

They had good agreement with the target for Chromium and Lead. They were biased high for Arsenic and Copper, and were slightly high for Cadmium in MET3. For Aluminum and Zinc, Laboratory 9412 was erratic, though in opposite quadrants of the Youden plots (Figures 1 and 8, respectively). For Aluminum, it appears that they may have a negative intercept, as they were biased low for the low sample, MET2. For Zinc, they appear to have a high intercept plus a low slope, as discussed in the overview, above. Comparison with international reference standards should help resolve the bias for Arsenic, Copper and Cadmium. Determining the source(s) of erratic performance for Aluminum and Zinc may require more involved investigations by this laboratory.

Laboratory 9413

The target level for Cadmium was below the detection limit of Laboratory 9413. They were the only participant to pretreat the samples before instrumental analysis.

Laboratory 9413 had good agreement with the target for Aluminum, Arsenic, and Zinc. They were biased high for Copper, as were all of the participants, but they were the closest to the target. They were biased low for Lead and Selenium. Comparison with international reference standards should resolve these differences. For Chromium, they were erratic, as they were high for MET2 but had good agreement with the target for MET3 (Figure 4).

Laboratory 9414

The target levels for Arsenic, Chromium, and Selenium, plus Copper in MET2, were below the detection limits of Laboratory 9414.

They were biased low for Aluminum and Lead. They were biased high for Cadmium in both samples and Copper in MET3. These differences may be resolved by comparison with international reference standards. For Zinc, they appear to have a high intercept plus a low slope, as discussed in the overview, above. More detailed investigation by Laboratory 9414 may be necessary to determine the source(s) of this problem. Further method development to lower detection limits for several elements is also recommended.

Laboratory 9415

Laboratory 9415 only reported results for four of the Trace Metals. They had good agreement with the target for Cadmium and Chromium. They were biased high for Arsenic and biased low for Lead. These differences may be resolved by comparison with international reference standards. They also had precision problems for Lead (Figure 6), which should be investigated, as this is the most important Trace Metal for the IADN program².

Laboratory 9416

Laboratory 9416 noted that their detection limit for Selenium is approximately 1.0 µg/L, and they were unable to detect the low levels in the study samples.

They had good agreement with the target for Arsenic, Cadmium, Chromium, Lead, and Zinc. They were biased low for Aluminum. They were biased high for Copper, as were all of the participants, but they were the highest away from the target. These differences may be resolved by comparison with international reference standards.

4.3 Part B: Overview of Interlaboratory Performance

Results were received from all of the laboratories who provided filters for this part of the study.

The interlaboratory mean, median, and standard deviation for the MET5 and MET6 results are included for information only. They do not give an accurate assessment of agreement with the target values, as three of the participants detected three or more of the elements in the unspiked filter, MET4. This suggests that the filters may have absorbed some Trace Metals, either during the spiking procedure or during transportation and therefore affecting the total result being reported for MET5 and MET6. The following discussion is based on the blank-corrected values for MET5 and MET6.

The Youden technique, as described in Section 4.1, was also used to plot the results from the spiked filters.

Laboratory 9411 had contamination problems for Aluminum, that are discussed in the individual laboratory review below. Their results were excluded from the statistical calculations and from the graph (Figure 9). The remaining three laboratories' results for Aluminum appear to differ primarily based on differences in standards. Laboratory 9416 was biased low.

The Arsenic results (Figure 10) suggest that the main difference between the laboratories is a difference in standards, as all the results are grouped close to the diagonal line. Comparison with reference standards should resolve these differences.

The Cadmium results (Figure 11) has the same pattern of results, close to the diagonal line, as the Arsenic results, except for Laboratory 9414, which was erratic. The same recommendation applies for this element as for Arsenic.

The Chromium results were very variable (Figure 12). Laboratory 9417 noted that they had a contamination problem with Chromium in their blank filters (Table 2A),

which contributed to their low results after the "blank" correction. Laboratory 9411 also appears to have a contamination problem (Table 2B).

Three of the participants had good agreement with each other and the target for Copper (Figure 13). Laboratory 9411 was biased low.

The Lead results (Figure 14) show two laboratories (9416 and 9414) having good agreement with each other and the target. Laboratory 9411 was biased low.

The Selenium results (Figure 15) demonstrate considerable spread among the participants. As all of the results are close to the diagonal line, indicating good within laboratory precision, the primary difference among the laboratories may be attributed to a difference in standards.

Three of the participants had Zinc results along the diagonal line (Figure 16), indicating good within laboratory precision, though they did not all agree with the target values. Laboratory 9414 had a high result for MET5, suggesting a possible intercept problem. This is discussed further below.

Each of the participants used a different analytical technique for the analysis of the filters (Table 4). Without some replication of technique among the participants, it is impossible to make an evaluation that indicates any advantages or disadvantages to any of these techniques.

Except for the Neutron Activation Analysis used by Laboratory 9417 (Table 4), the other participants required a preparation step for the analysis of the filters. This may introduce more variability between the laboratories as compared to the precipitation analysis which didn't require a preparation step (except for Laboratory 9413, Table 3). However this was not always the case. The between-laboratory variability was similar between Parts A and B for Aluminum and Arsenic. There was less between-laboratory variability in Part B for Cadmium and Lead than in Part A, despite the preparation step. There was greater between-laboratory variability in Part B than Part A for Chromium and Zinc. For Copper, there was less variability for the lower concentration sample in Part A (precipitation) but for the higher concentration sample, between-laboratory variability was less in Part B (spiked filter).

4.4 Part B: Individual Laboratory Performance

As was noted above in Section 4.2, the individual laboratory review is based only on the results of this study, with no comparisons to any previous studies.

Laboratory 9411

Laboratory 9411 used a 4X dilution to obtain results for Chromium in all three samples, for Copper in MET6, and for Lead in MET5 and MET6.

They appear to have had a contamination problem with Aluminum, as they reported very high values for all three samples. Their results, even after subtracting the MET4 value from MET5 and MET6, were biased very high, and were excluded from the statistical calculations. While they were biased slightly high for Aluminum in the precipitation samples (Table 1A), it was small relative to the bias in the filters (Table 2A). Laboratory 9411 should carefully investigate their

preparation procedures, as this appears to be the most probable source of the contamination.

They had good agreement with the target for Zinc. They were biased low for Arsenic, Cadmium, Copper, Lead, and Selenium. They were biased high for Chromium. The problem for Cadmium appears standard related, as they were also biased high for MET3 in the precipitation analysis. However for the other Trace Metals, they were biased high in the precipitation analysis. Since there were no preparation steps for the precipitation analysis, but there were for the filter analysis, it appears that their digestion procedure is under-recovering many of the Trace Metals. Laboratory 9411 should undertake some recovery studies to determine if their procedure is fully recovering the Trace Metals of interest.

Laboratory 9414

Laboratory 9414 had good agreement with the target for Aluminum, Arsenic, Chromium, Copper, Lead, and Selenium. They had erratic performance for Cadmium (Figure 11), the source of which is more difficult to determine. As noted in the Overview (Section 4.3), they had a high Zinc result for MET5, suggesting a possible intercept problem. This was similar to their performance for Zinc for the precipitation analysis.

Laboratory 9416

Laboratory 9416 had good agreement with the target for Arsenic, Cadmium, Chromium, and Lead. They were biased low for Aluminum and Copper, and were biased high for Selenium and Zinc. For all parameters they demonstrated good within laboratory precision. Their low results for Copper may be due to their sample preparation procedure, as they were biased high for Copper in the precipitation analysis, for which there was no preparation procedure. Further investigations are needed to determine if this is the source of the low bias.

Laboratory 9417

Laboratory 9417 noted that Chromium was contaminated in the blank filters. They did not perform any sample preparation procedures. However they did cut the spiked filters into two pieces prior to analysis, analyze them separately, and then sum the results. Only the summed results were reported.

They had good agreement with the target for Arsenic and Copper. Their result for Zinc in MET5 had good agreement with the target, though they were low for MET6. They were biased low for Aluminum, Cadmium, and Selenium. Their problems with Chromium on the blank filters (MET4A and MET4B) were noted above, and after the blank correction was done, resulted in very low results (Figure 12).

5 CONCLUSION

The overall interlaboratory performance in Part A (precipitation) of this study has shown improvements (Aluminum and Chromium) or a maintenance of performance from the previous studies^{4,5}, except for Selenium. Each laboratory had individual problems with different Trace Metals. Comparison with reference materials should help improve some of these problems.

The low target levels for many of these Trace Metals were beyond the analytical capability of several of the participants. These levels were similar to mean precipitation values reported by the National Water Research Institute⁷. As the IADN network is a research program, the lowest possible levels of detection are to be desired from the laboratories providing data. Further method development to improve detection capabilities and to improve between-laboratory variability at these low concentrations is recommended.

The analysis of the spiked filters in Part B was the first time the interlaboratory performance had been compared among these laboratories. While there were individual problems with some of the parameters, the differences among the laboratories were not extreme. Some of the differences may be attributable to differences in the filter preparation procedures and could be the basis for further studies. Future intercomparisons should help improve the comparability among these laboratories.

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7. Strachan, W.M.J. and Burniston, D.; 1994. *NWRI Precipitation Sampling IADN 1990-1991.* National Water Research Institute, Burlington, Ontario, presented at the 1994 IADN Workshop, June 4-5, 1994, Windsor, Ontario, Canada.

7 APPENDIX 1 - RESULTS AND GRAPHS

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TABLE 1 - INTERLABORATORY STUDY 94-1, PART A
SPIKED PRECIPITATION
RESULTS IN µg/L

ELEMENT	TARGET	9411	9412	9413	9414	9415	9416	MEAN	MEDIAN	STD DEV	%RSD	n
MET1												
Al	-	5.1	N/A	2	7.0		1.51					
As	-	<0.5	<0.10	<0.1	<1.0	0.077	ND					
Cd	-	0.08	<0.10	<0.1	<0.2	0.083	0.05					
Cr	-	<0.5	<0.10	<0.2	<2.0	0.040	ND					
Cu	-	2.1	1.2	1.5	<2.0		0.85					5
Pb	-	0.3	<0.10	<0.2	0.2	0.315	0.13					
Se	-	<0.5	N/A	0.1	<1.0		N/A					
Zn	-	3.6	3.4	2.4	4.0		2.82					
MET2												
Al	25	32.7	21	26	24		21.71	25.08	24	4.69	18.71%	5
As	0.10	<0.5	0.13	0.1	<1.0	0.203	0.11	0.14	0.12	0.047	34.28%	4
Cd	0.07	0.19	<0.10	<0.1	0.2	0.141	0.12	0.16	0.165	0.038	23.61%	4
Cr	0.50	0.5	0.50	0.6	<2.0	0.498	0.51	0.52	0.50	0.044	8.45%	5
Cu	0.52	2.8	2.0	2.2	<2.0		1.85	2.21	2.1	0.42	18.85%	4
Pb	0.75	1.2	0.68	0.4	0.6	0.513	0.87	0.71	0.64	0.288	40.48%	6
Se	0.10	0.5	N/A	0.1	<1.0		N/A	0.30				2
Zn	3.75	6.7	8.0	6.2	8.5		6.99	7.28	6.99	0.95	13.02%	5
MET3												
Al	100	123.2	104	101	79		94.28	100.3	101	16.04	15.99%	5
As	0.40	<0.5	0.48	0.4	<1.0	0.550	0.39	0.46	0.44	0.075	16.50%	4
Cd	0.30	0.53	0.35	<0.1	0.5	0.386	0.37	0.36	0.378	0.189	53.13%	6
Cr	2.00	2.7	1.9	1.9	<2.0	1.931	2.03	2.09	1.931	0.344	16.44%	5
Cu	2.10	4.7	3.8	3.8	3.0		3.42	3.74	3.8	0.63	16.78%	5
Pb	3.00	4.1	3.1	2.5	2.1	2.910	3.16	2.98	3.005	0.680	22.83%	6
Se	0.40	1.2	N/A	0.3	<1.0		N/A	0.75				2
Zn	15.0	17.4	17	17.2	17.0		19.33	17.59	17.2	0.99	5.62%	5

TABLE 1 - INTERLABORATORY STUDY 94-1, PART A
SPIKED PRECIPITATION
RESULTS IN $\mu\text{g/L}$

ELEMENT	TARGET	9411	9412	9413	9414	9415	9416	MEAN	MEDIAN	STD DEV	%RSD	n
MET2-MET1												
Al	25	27.6	21	24	17		20.2	21.96	21	4.02	18.30%	5
As	0.10	<0.5	0.13	0.1	<1.0	0.126	0.11	0.12	0.118	0.014	12.01%	4
Cd	0.07	0.11	<0.1	<0.1	0.2	0.058	0.07	0.11	0.09	0.064	58.72%	4
Cr	0.50	0.5	0.5	0.6	<2.0	0.458	0.51	0.51	0.5	0.052	10.18%	5
Cu	0.52	0.7	0.8	0.7	<2.0		1.00	0.80	0.75	0.14	17.68%	4
Pb	0.75	0.9	0.68	0.4	0.4	0.198	0.74	0.55	0.54	0.262	47.46%	6
Se	0.10	0.5	N/A	0	<1.0		N/A	0.25				2
Zn	3.75	3.1	4.6	3.8	4.5		4.17	4.03	4.17	0.61	15.09%	5
MET3-MET1												
Al	100	118.1	104	99	72		92.77	97.2	99	16.89	17.38%	5
As	0.40	<0.5	0.48	0.4	<1.0	0.473	0.39	0.44	0.436	0.047	10.86%	4
Cd	0.30	0.45	0.35	0	0.5	0.303	0.32	0.32	0.335	0.175	54.56%	6
Cr	2.00	2.7	1.9	1.9	<2.0	1.891	2.03	2.08	1.9	0.349	16.75%	5
Cu	2.10	2.6	2.6	2.3	3.0		2.57	2.61	2.6	0.25	9.56%	5
Pb	3.00	3.8	3.1	2.5	1.9	2.595	3.03	2.82	2.813	0.645	22.88%	6
Se	0.40	1.2	N/A	0.2	<1.0		N/A	0.70				2
Zn	15.0	13.8	13.6	14.8	13.0		16.51	14.34	13.8	1.37	9.58%	5

TABLE 2 - INTERLABORATORY STUDY 94-1, PART B
SPIKED FILTERS
RESULTS IN µg/Filter

ELEMENT	TARGET	9411	9414	9416	9417	MEAN	MEDIAN	STD DEV	%RSD	n	
MET4											
					MET4A	MET4B	MEAN #				
Al	-	1175	>1.3	ND	2.82±0.05	2.64±0.03	2.73				
As	-	0.24	>0.05	ND	0.0015±0.0002	0.0013±0.0005	0.0014				
Cd	-	0.006	>0.03	ND	0.017±0.003	0.013±0.002	0.015				
Cr	-	1.38	>0.25	0.004	2.00±0.02	1.78±0.02	1.89				
Cu	-	0.47	>0.20	0.002	0.36±0.03	0.34±0.02	0.35				
Pb	-	0.34	>0.20	ND	N/A	N/A	N/A				
Se	-	0.03	>0.05	ND	<0.014	<0.014	<0.014				
Zn	-	13.1	>0.25	0.013	3.79±0.06	1.62±0.04	2.705				
MET5											
Al	16	1263	17.5	12.230	17.00±0.14		15.58 *	17.00 *	2.909 *	18.7% *	3
As	1.6	1.4	1.61	1.702	1.52±0.01		1.558	1.565	0.129	8.3%	4
Cd	0.12	0.09	0.09	0.124	0.112±0.008		0.104	0.101	0.017	16.2%	4
Cr	0.48	1.96	0.55	0.436	1.81±0.02		1.189	1.18	0.807	67.9%	4
Cu	2.52	1.12	2.56	2.280	2.76±0.06		2.18	2.42	0.734	33.7%	4
Pb	1.60	1.61	1.70	1.512	N/A		1.607	1.61	0.094	5.8%	3
Se	0.40	0.14	0.42	0.560	0.335±0.009		0.364	0.3775	0.176	48.3%	4
Zn	5.60	18.8	10.0	7.217	8.20±0.11		11.05	9.1	5.291	47.9%	4
MET6											
Al	40	1538	38.6	29.645	36.56±0.31		34.93 *	36.56 *	4.694 *	13.4% *	3
As	4.0	3.63	3.79	4.269	3.52±0.02		3.802	3.71	0.330	8.7%	4
Cd	0.29	0.23	0.32	0.313	0.282±0.008		0.286	0.2973	0.041	14.3%	4
Cr	1.20	3.49	1.27	1.214	2.63±0.03		2.151	1.95	1.107	51.5%	4
Cu	6.29	5.63	6.07	5.702	5.93±0.13		5.833	5.816	0.203	3.5%	4
Pb	4.00	3.55	3.75	3.933	N/A		3.744	3.75	0.191	5.1%	3
Se	1.00	0.36	0.95	1.245	0.761±0.011		0.829	0.8555	0.371	44.7%	4
Zn	14.00	27.5	14.2	16.537	14.95±0.17		18.3	15.743	6.212	34.0%	4

TABLE 2 - INTERLABORATORY STUDY 94-1, PART B
SPIKED FILTERS
RESULTS IN µg/Filter

ELEMENT	TARGET	9411	9414	9416	9417	MEAN	MEDIAN	STD DEV	%RSD	n
METS-MET4										
Al	16	88	17.5	12.230	14.27	14.66 *	15.885 *	2.657 *	18.1% *	3
As	1.6	1.16	1.61	1.702	1.5186	1.497	1.5643	0.237	15.8%	4
Cd	0.12	0.084	0.09	0.124	0.097	0.098	0.0935	0.018	17.9%	4
Cr	0.48	0.58	0.55	0.432	-0.08	0.370	0.491	0.307	82.9%	4
Cu	2.52	0.65	2.56	2.278	2.41	1.974	2.344	0.890	45.1%	4
Pb	1.60	1.27	1.70	1.512	N/A	1.494	1.512	0.216	14.4%	3
Se	0.40	0.11	0.42	0.560	0.335	0.356	0.3775	0.188	52.9%	4
Zn	5.60	5.7	10.0	7.204	5.495	7.099	6.452	2.078	29.3%	4
MET6-MET4										
Al	40	363	38.6	29.645	33.83	32.33 *	36.215 *	4.481 *	13.2% *	3
As	4.0	3.39	3.79	4.269	3.5186	3.741	3.6543	0.389	10.4%	4
Cd	0.29	0.224	0.32	0.313	0.267	0.280	0.2897	0.045	15.9%	4
Cr	1.20	2.11	1.27	1.210	0.74	1.332	1.2397	0.570	42.8%	4
Cu	6.29	5.16	6.07	5.700	5.58	5.627	5.64	0.375	6.7%	4
Pb	4.00	3.21	3.75	3.933	N/A	3.631	3.75	0.376	10.3%	3
Se	1.00	0.33	0.95	1.245	0.761	0.821	0.8555	0.383	46.7%	4
Zn	14.00	14.4	14.2	16.524	12.245	14.34	14.3	1.749	12.2%	4

* Laboratory 9411's results were excluded from the statistical calculations for Aluminum.
Laboratory 9417 requested two blank filters (MET4A and MET4B). A mean has been calculated from the two filters and used for the blank correction calculations.

TABLE 3 - PART A: METHODOLOGY OF PARTICIPANTS

PARTICIPANT	SAMPLE CONTAINER	PRESERVATIVE	SAMPLE PRE-TREATMENT	INSTRUMENTATION
9411	PET	Nitric Acid (Trace Metal Grade), 0.2% (v/v) when samples received	None	Perkin-Elmer GFAAS, Model PE4100ZL
9412	Teflon	None	None	ICP-MS, Perkin Elmer Elan 5000
9413	Teflon	As & Se: None Others: Pre-charged with 50% Nitric Acid Solution (2 mL/L)	As & Se: 4X conc., pre-digestion, Hydride formation Others: 10X concentration using USN	As & Se: ICP-OES, IRIS Others: ICP-OES, ARL 3580
9414	Teflon	Pre-charged with concentrated Nitric Acid	None	Al, Cr, Cu, Pb & Zn: ICP-MS and ICP-AES As, Se & Cd: GFAAS
9415	PET	0.25% Nitric Acid when samples received	None	AA, Perkin Elmer 5000
9416	PET	None	None	ICP-MS

Legend

AA	Atomic Absorption
GFAAS	Graphite Furnace Atomic Absorption Spectrophotometer
ICP-AES	Inductively Coupled Plasma - Absorption Emission Spectrophotometer
ICP-MS	Inductively Coupled Plasma - Mass Spectrometer
ICP-OES	Inductively Coupled Plasma - Optical Emission Spectrophotometer
USN	Ultra Sonic Nebulizer

TABLE 4 - PART B: METHODOLOGY OF PARTICIPANTS

PARTICIPANT	FILTER TYPE	PORTION OF FILTER SPIKED	SAMPLE PRE-TREATMENT	INSTRUMENTATION
9411	Gelman Type A/E Fibre	2.5" X 4"	1. Digestion with 10 mL aqua regia 25% (v/v) 2. Dilution to 50 mL with HPLC water	Al: ICP, Jarrell-Ash 975 As, Cd, Cr, Cu, Pb, Se: Perkin-Elmer GFAAS, Model PE4100ZL Zn: AA, PE2380
9414	47 mm Cellulose Ester	All	Digestion with concentrated Nitric Acid	Al, Cr, Cu, & Zn: ICP-AES Cd & Pb: GFAAS As & Se: HGAA
9416	47 mm Whatman 40	All	Extraction with 50 mL DDW	ICP-MS
9417	Whatman 41	2.5" X 8"	None (Sample cut into 2 pieces, analyzed separately and total of both pieces was reported)	NAA

Legend

AA	Atomic Absorption
GFAAS	Graphite Furnace Atomic Absorption Spectrophotometer
HGAA	Hydride Generation Atomic Absorption
ICP-AES	Inductively Coupled Plasma - Absorption Emission Spectrophotometer
ICP-MS	Inductively Coupled Plasma - Mass Spectrometer
ICP-OES	Inductively Coupled Plasma - Optical Emission Spectrophotometer
NAA	Neutron Activation Analysis
USN	Ultra Sonic Nebulizer

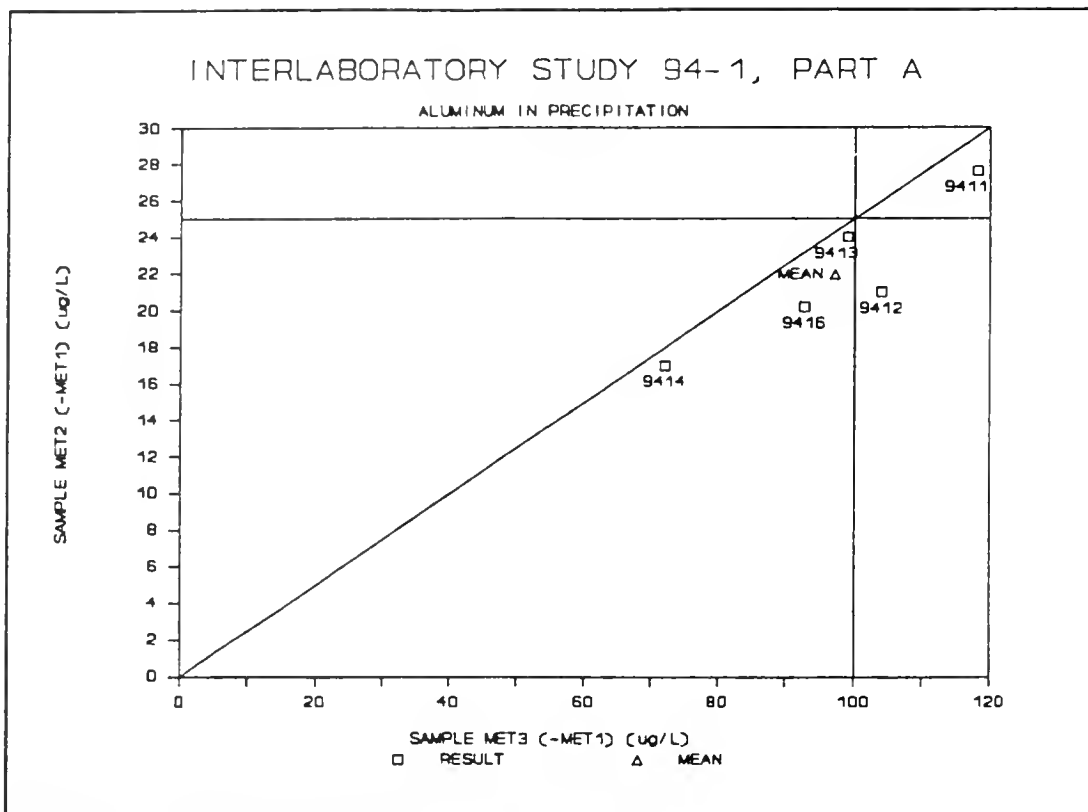


Figure 1 - Aluminum in Spiked Precipitation

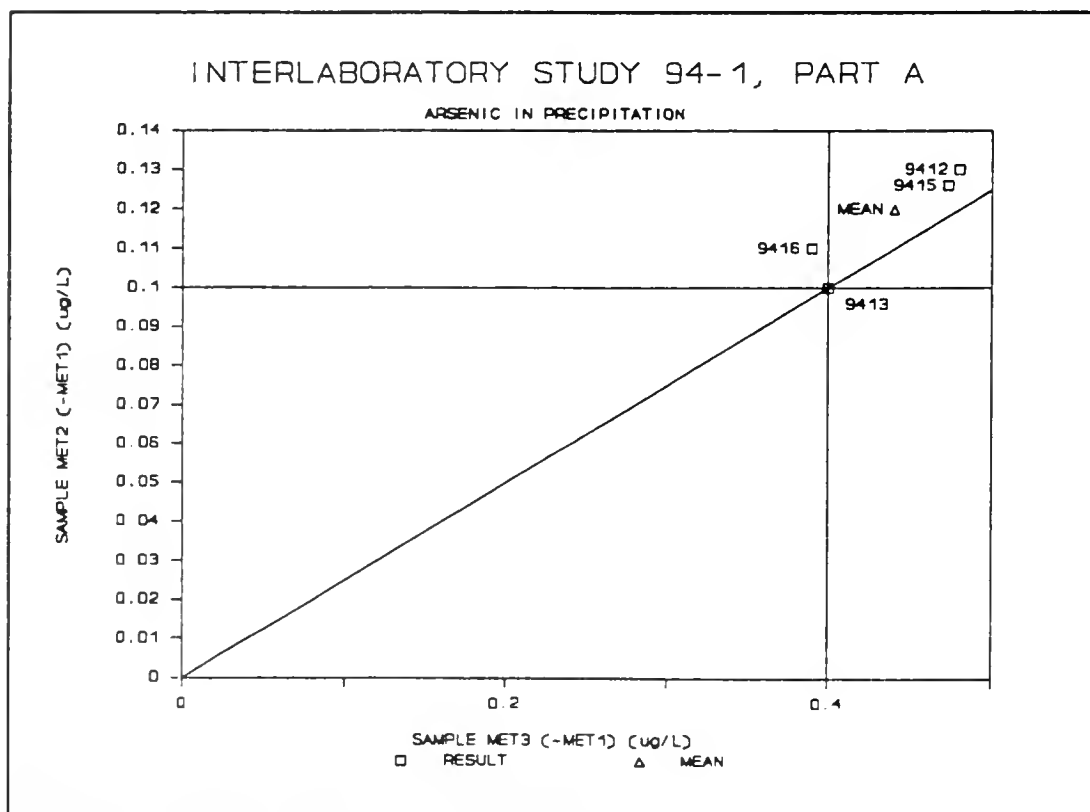


Figure 2 - Arsenic in Spiked Precipitation

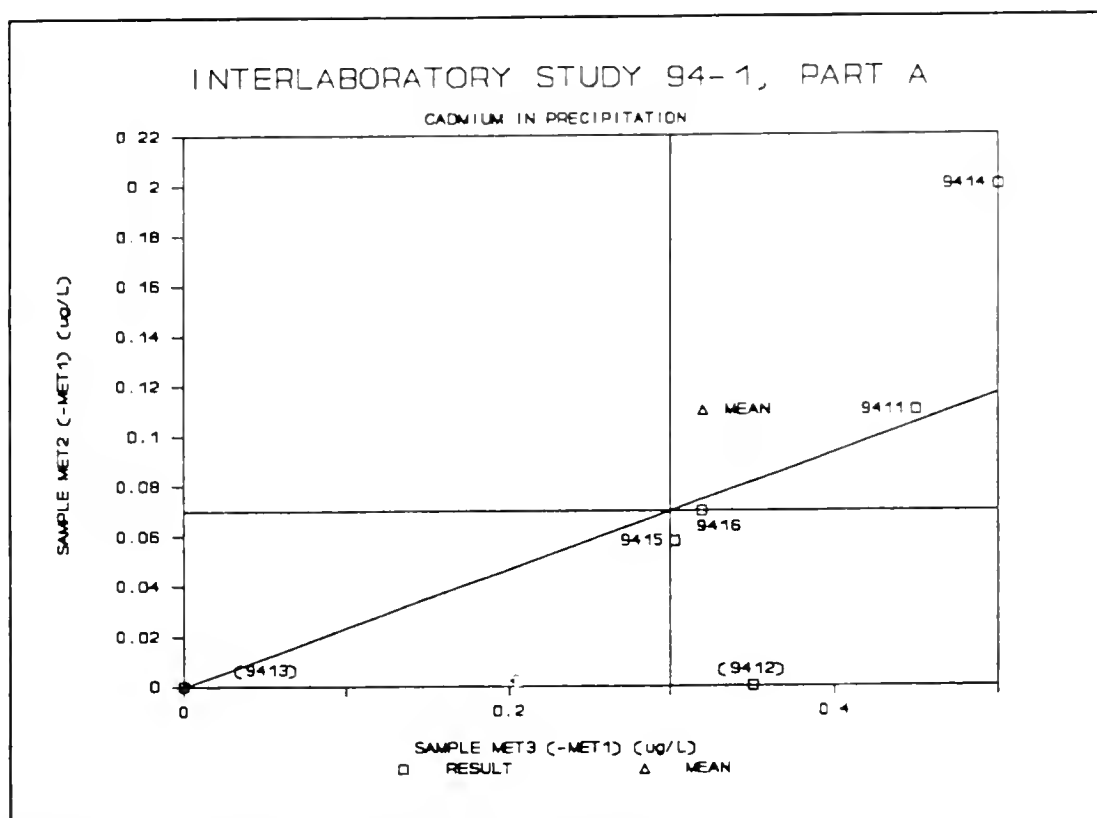


Figure 3 - Cadmium In Spiked Precipitation

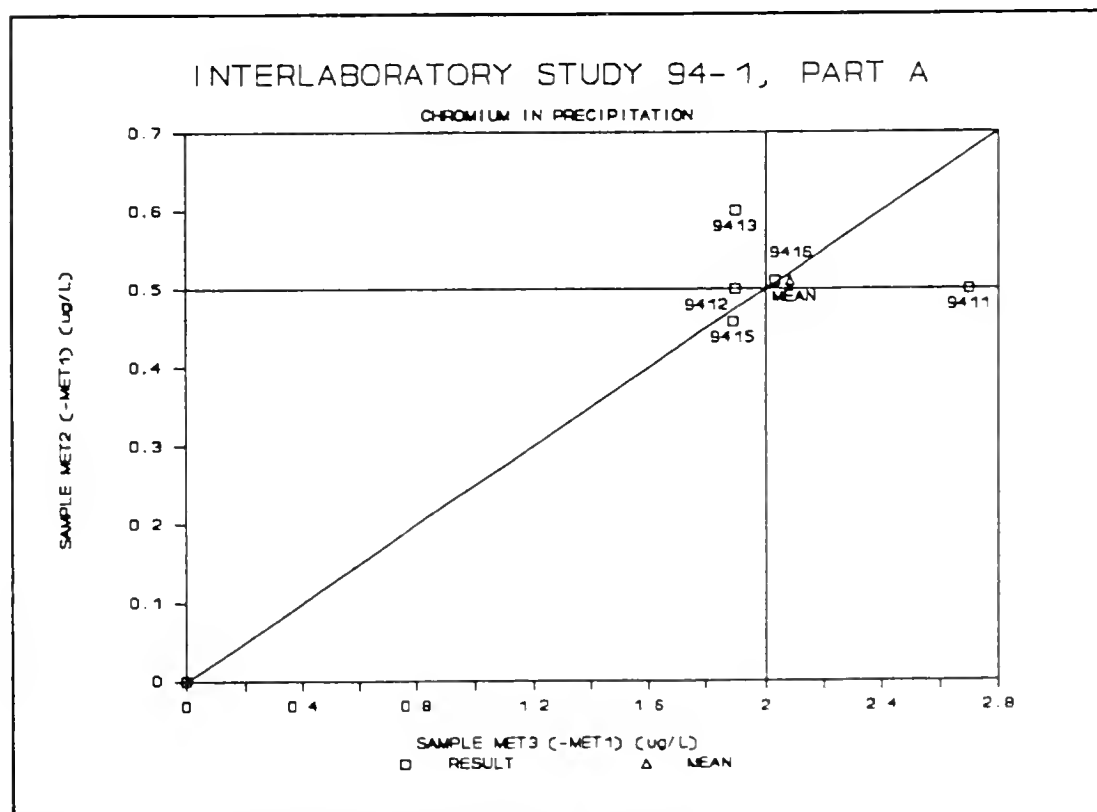


Figure 4 - Chromium In Spiked Precipitation

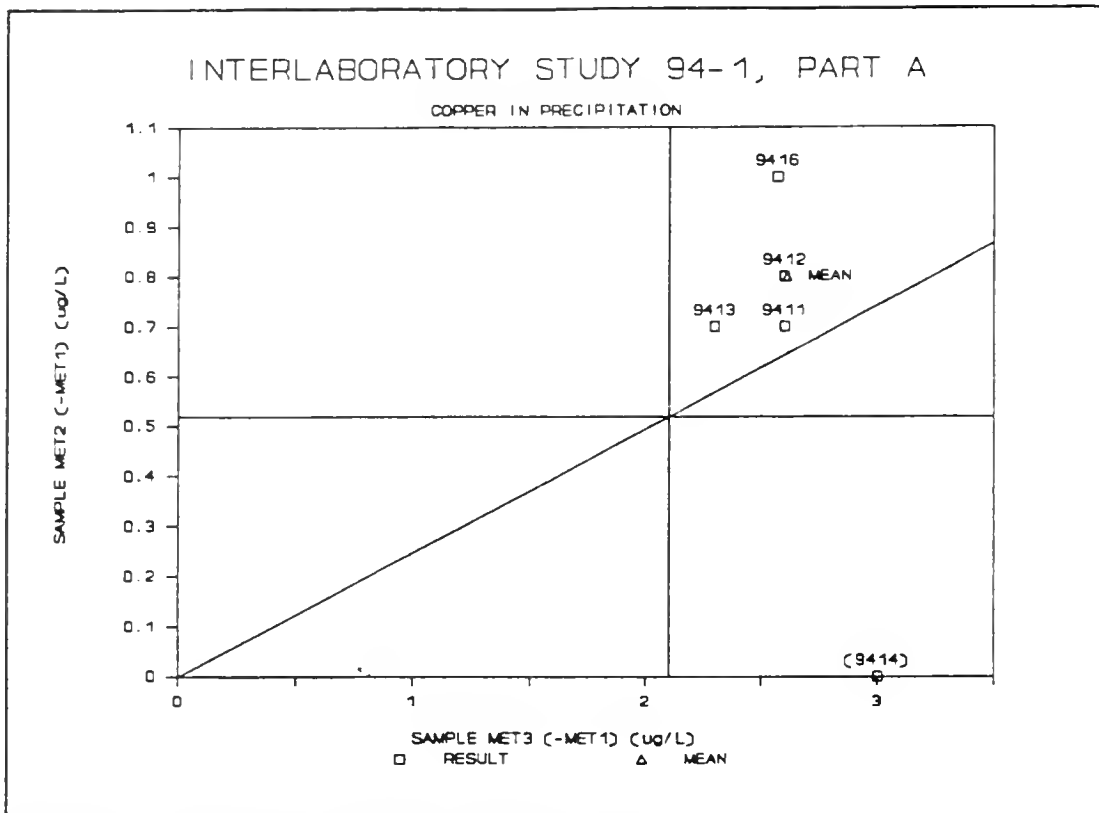


Figure 5 - Copper in Spiked Precipitation

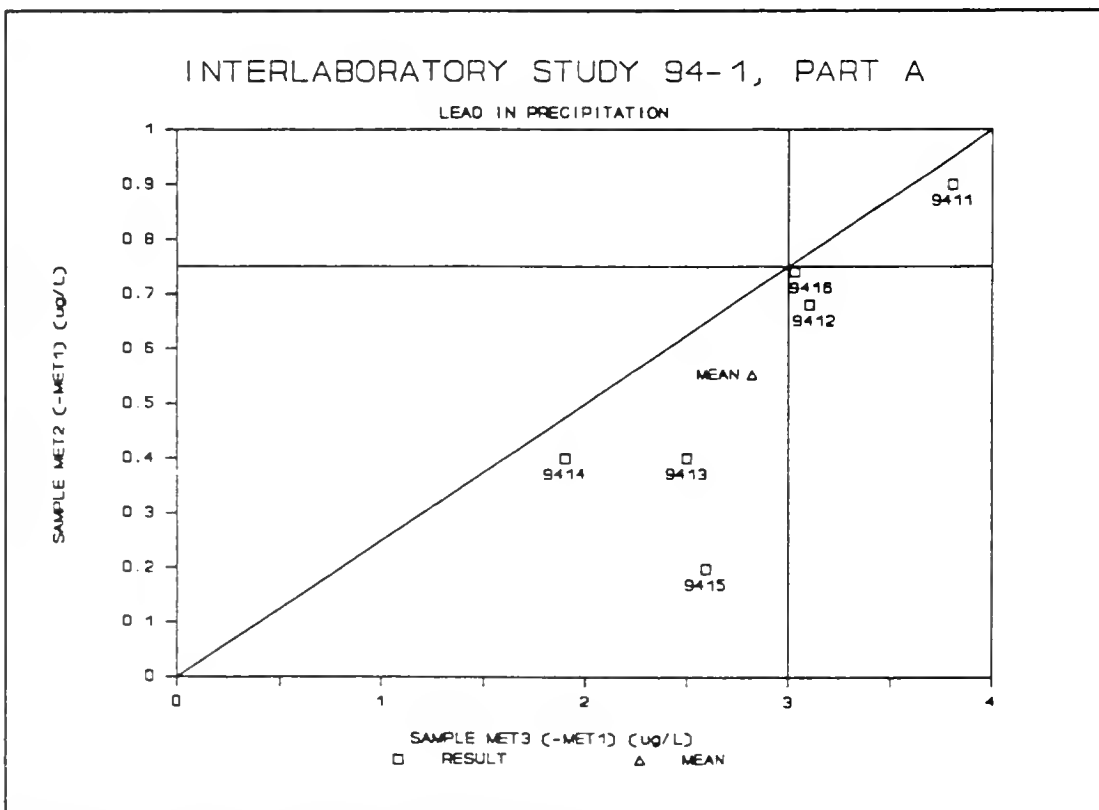


Figure 6 - Lead in Spiked Precipitation

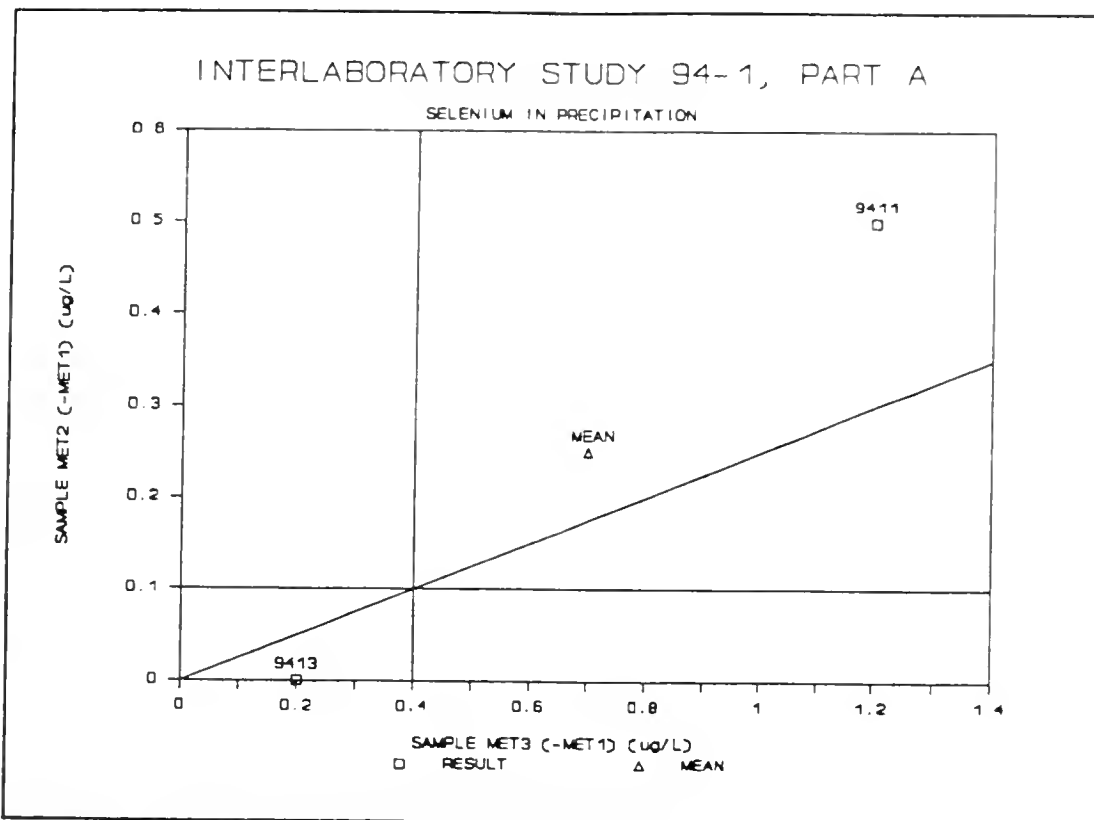


Figure 7 - Selenium in Spiked Precipitation

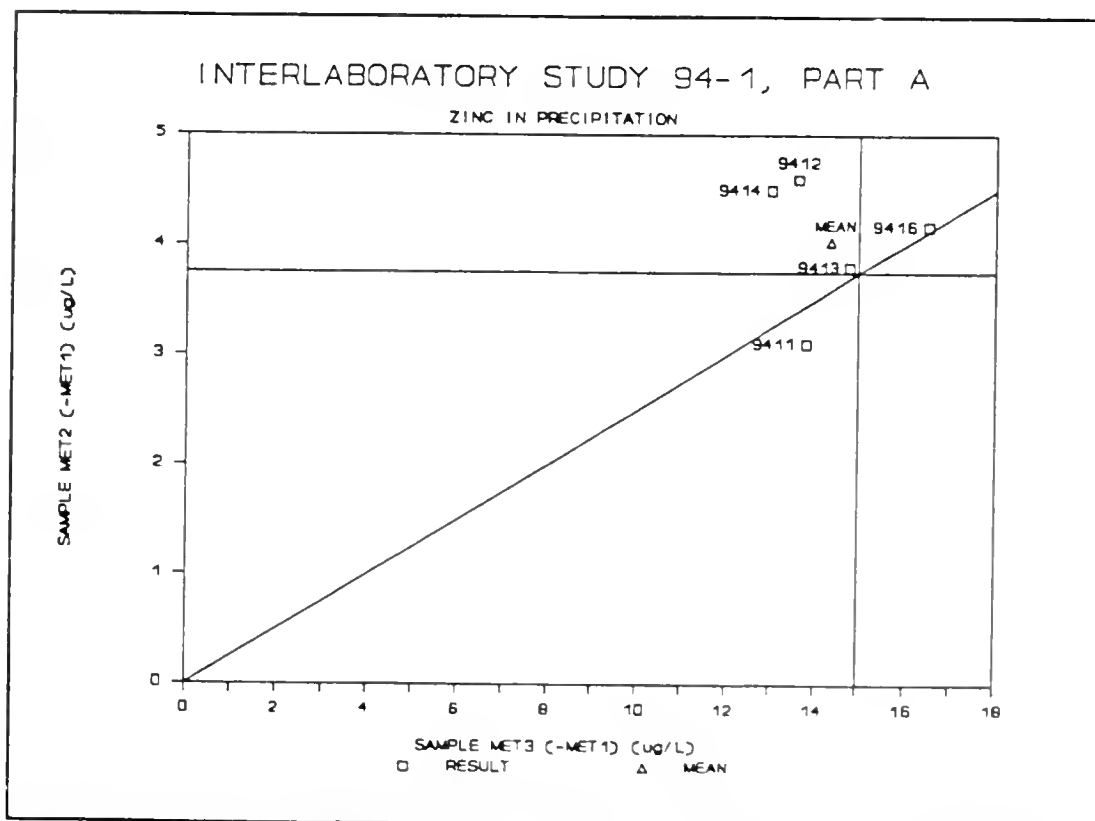


Figure 8 - Zinc in Spiked Precipitation

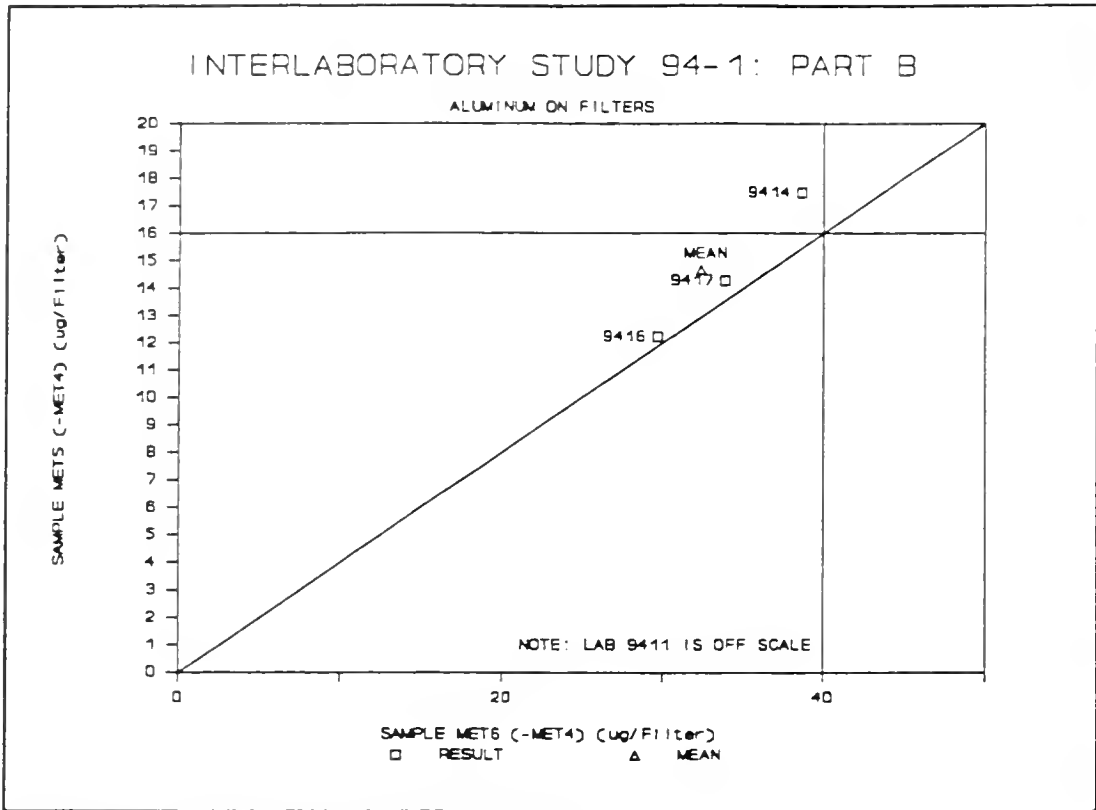


Figure 9 - Aluminum in Spiked Filters

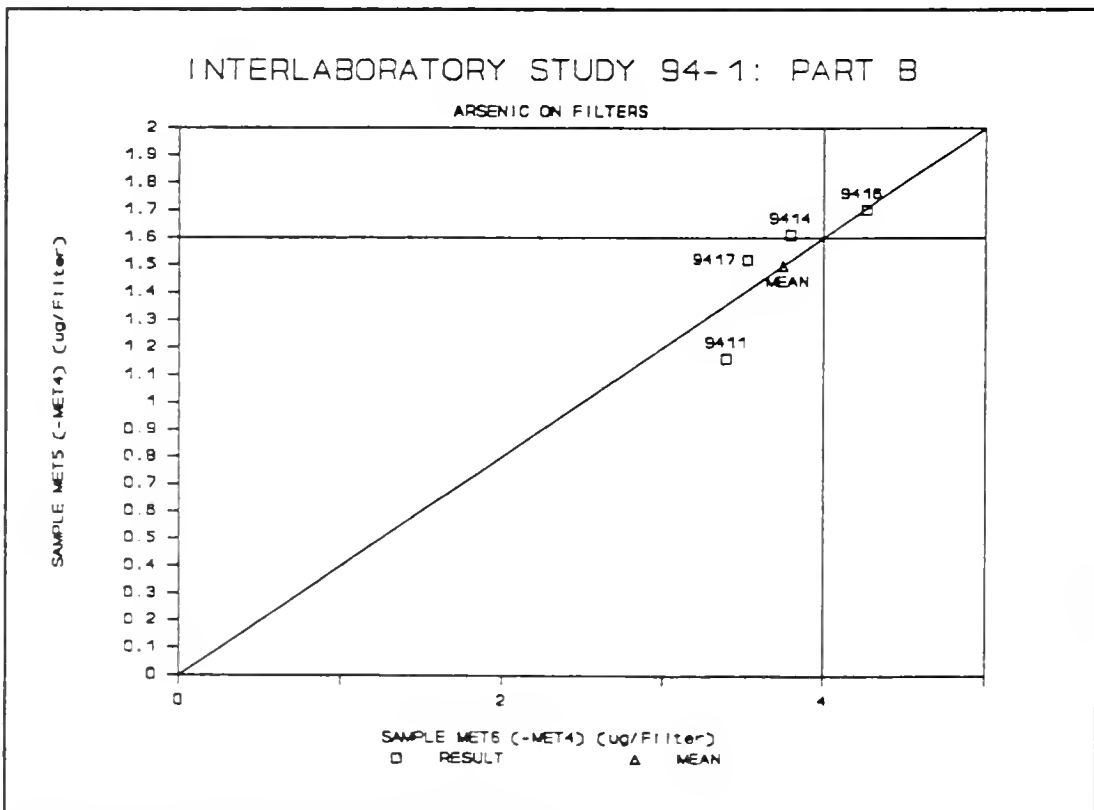


Figure 10 - Arsenic in Spiked Filters

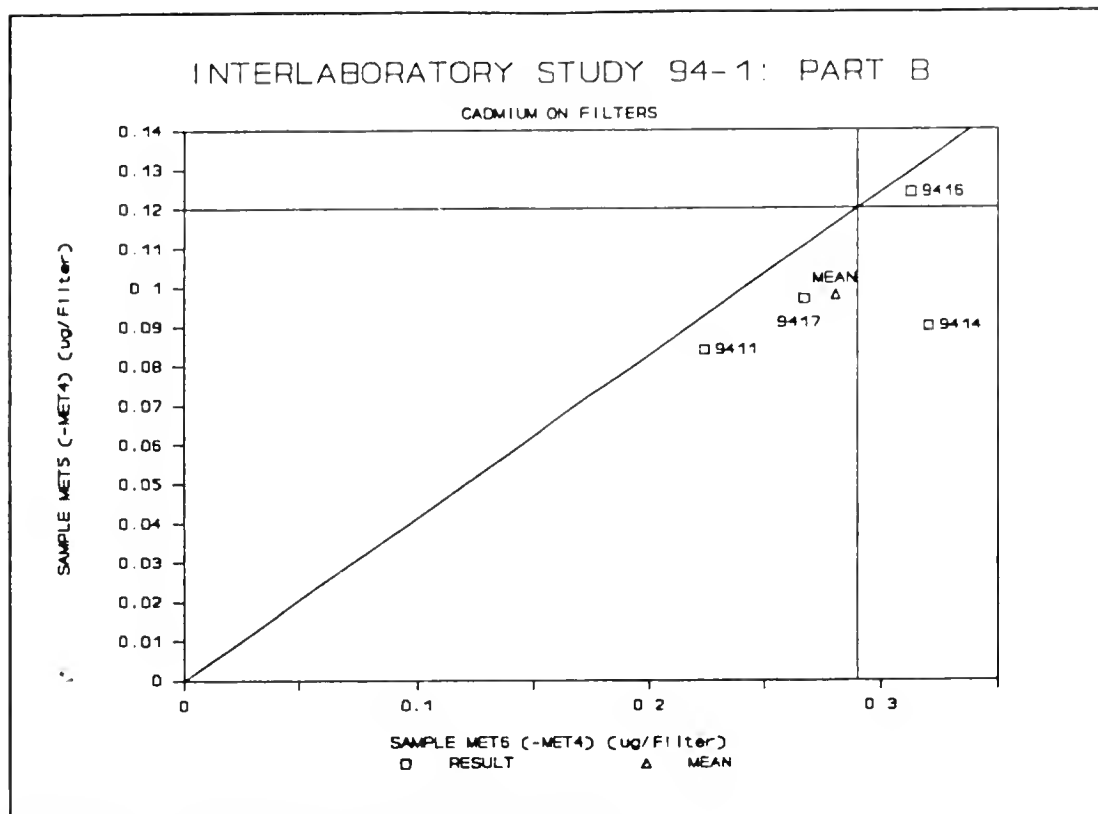


Figure 11 - Cadmium in Spiked Filters

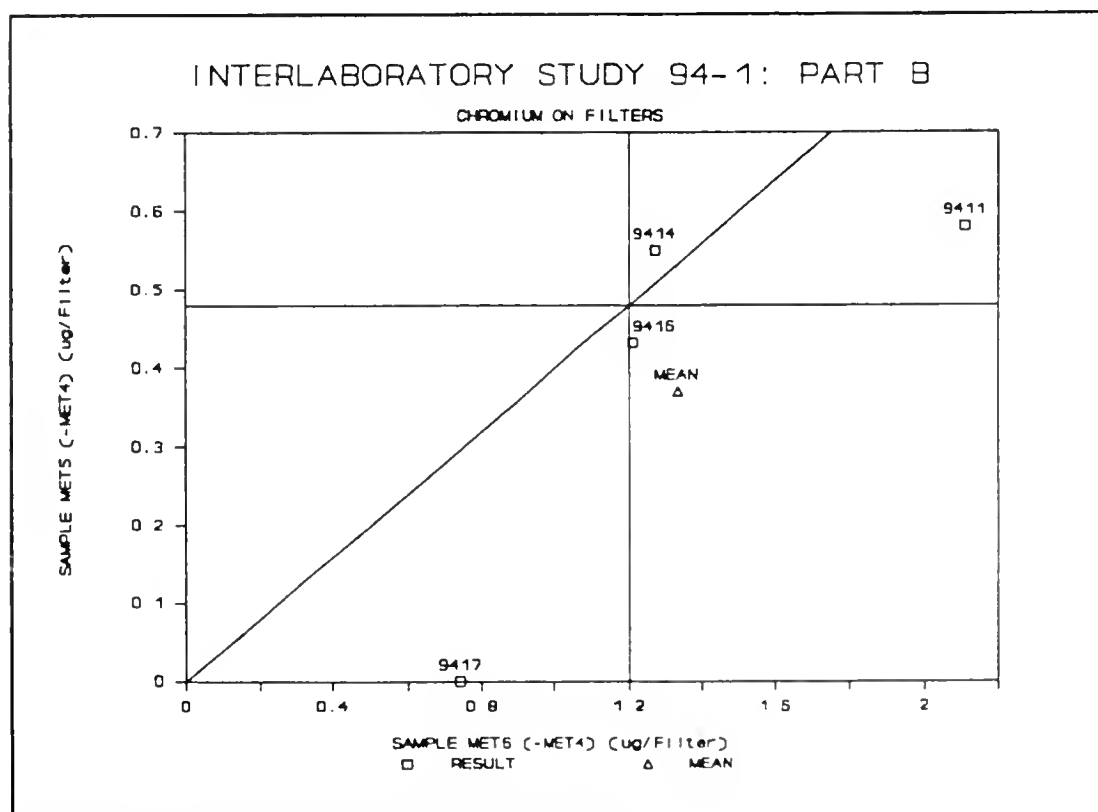


Figure 12 - Chromium in Spiked Filters

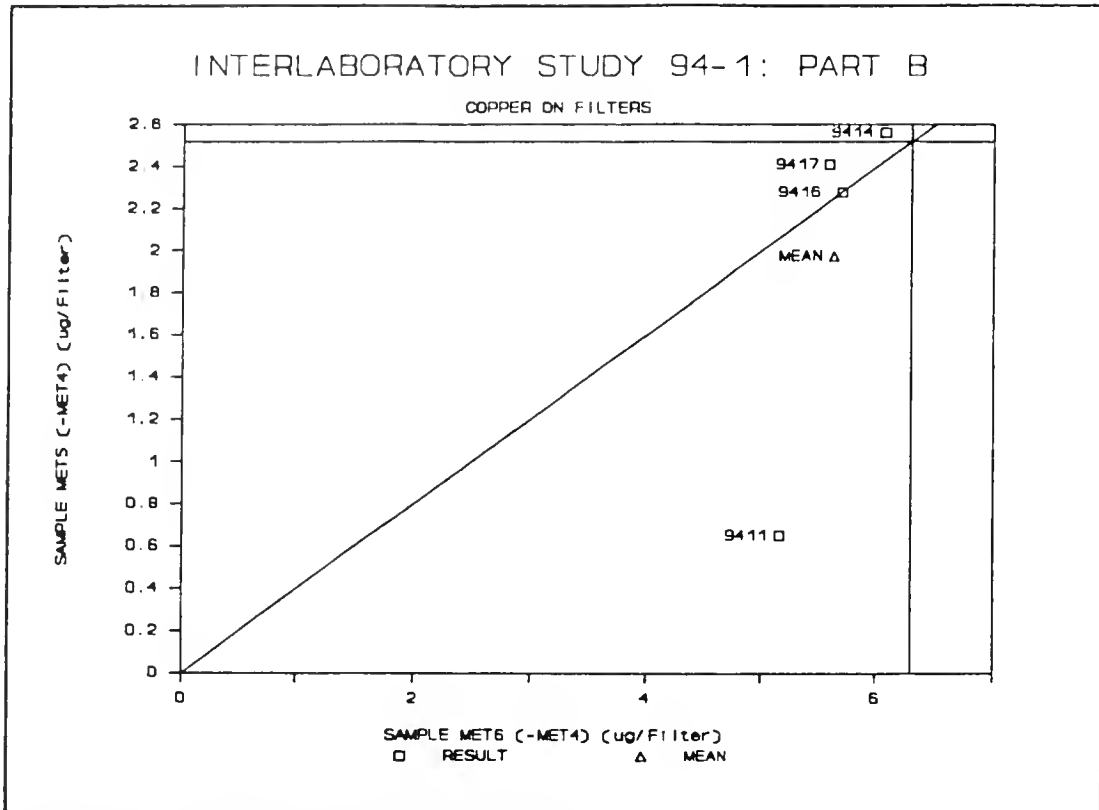


Figure 13 - Copper in Spiked Filters

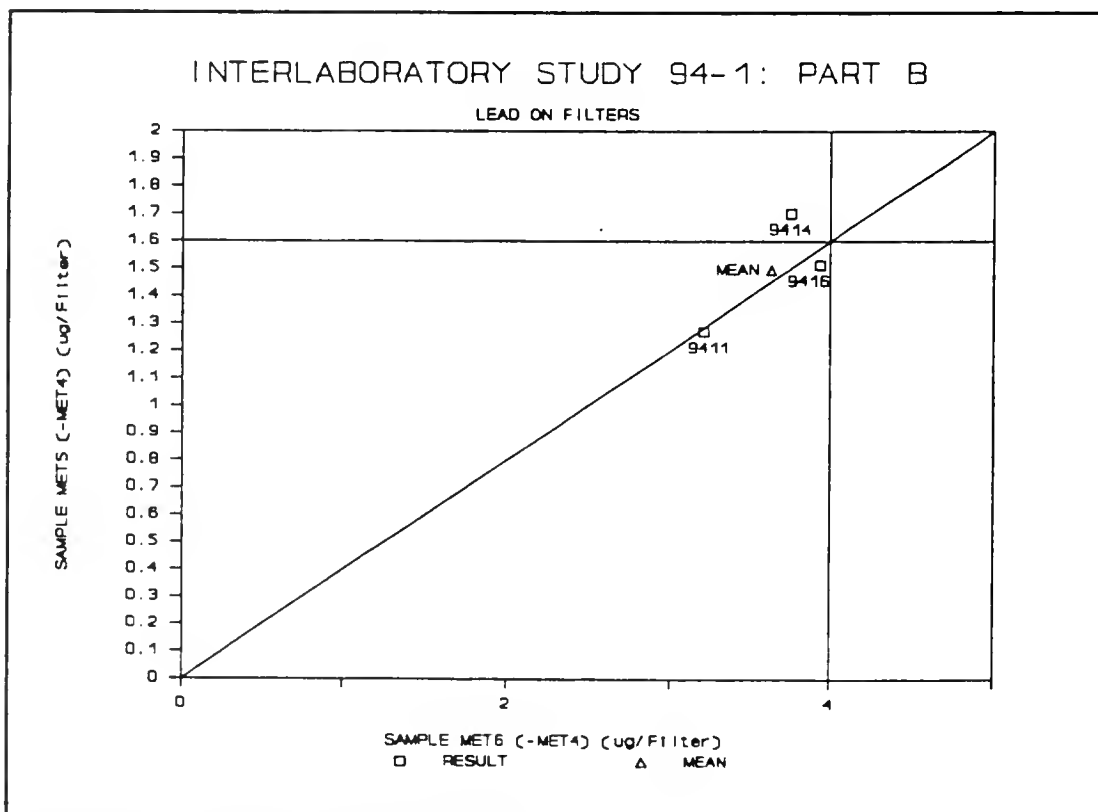


Figure 14 - Lead in Spiked Filters

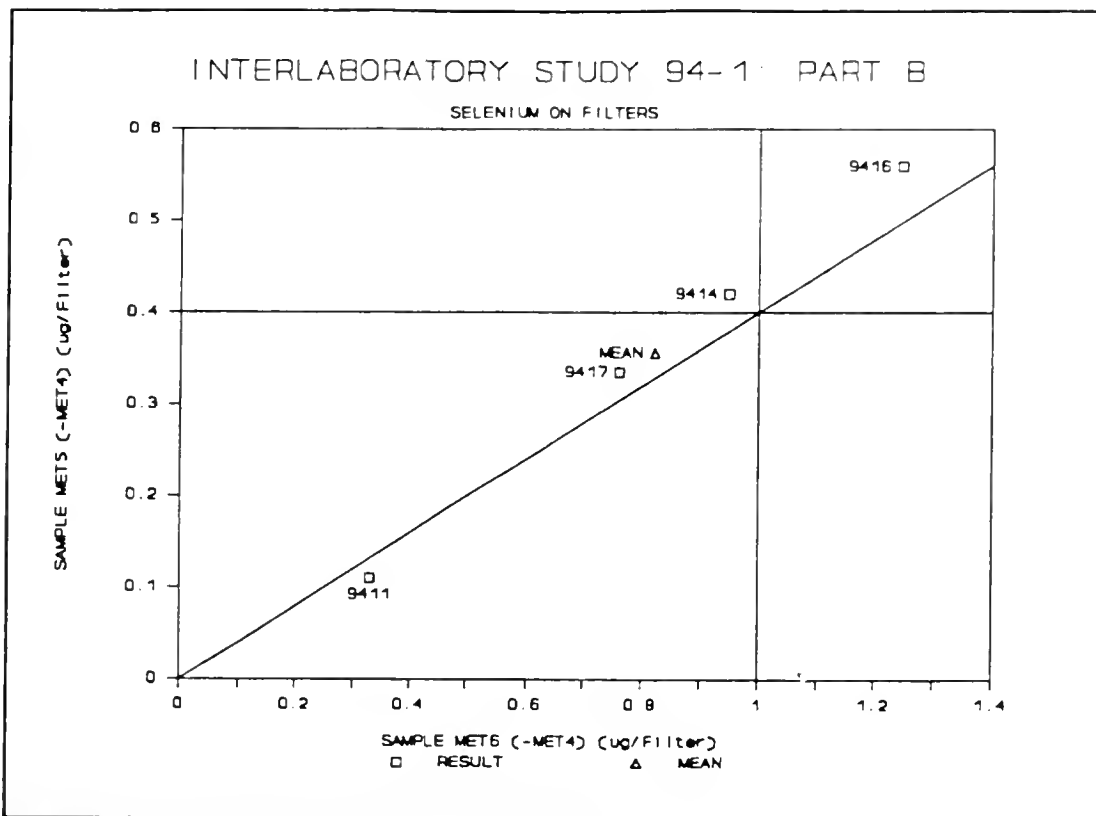


Figure 15 - Selenium in Spiked Filters

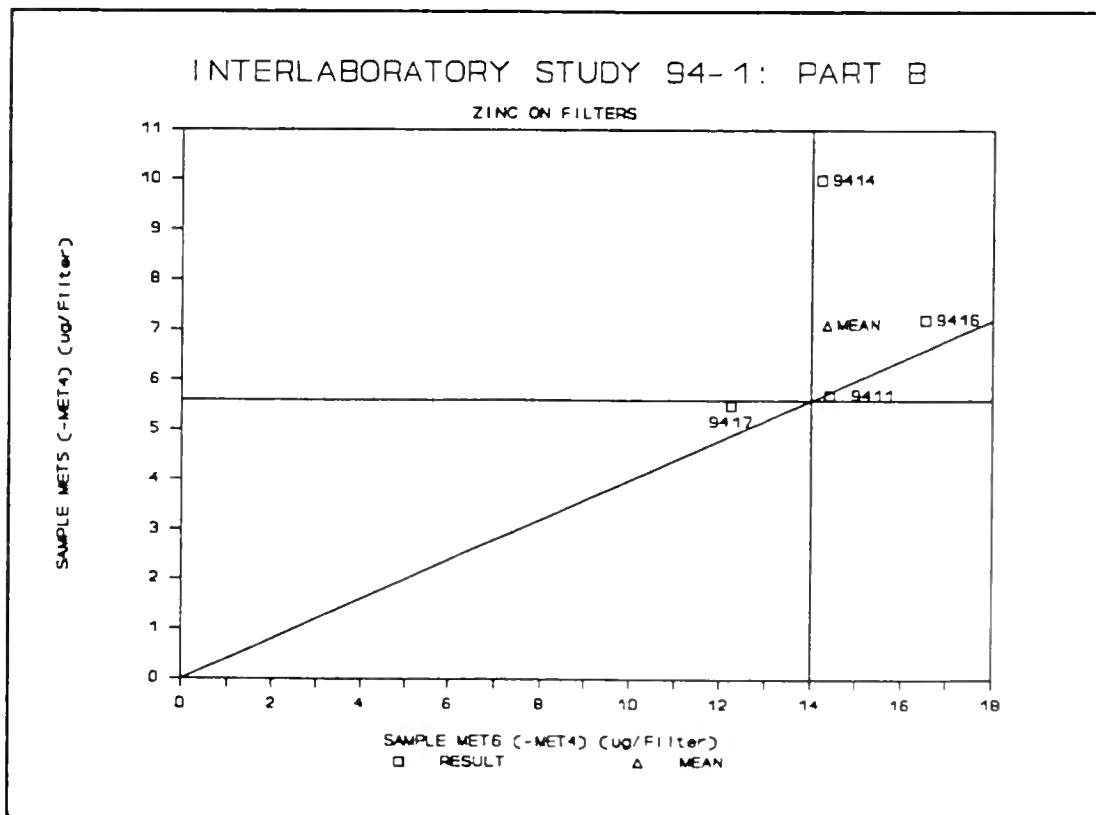


Figure 16 - Zinc in Spiked Filters

8 APPENDIX 2 - PARTICIPANTS AND CORRESPONDENCE

List of Participants

Debbie Burniston
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61801-2984
(217) 333-2486/1568

125 Resources Rd.
Etobicoke, Ontario, M9P 3V6
Phone: (416) 235-5842
FAX: (416) 235-5744

October 13, 1994

Dear Participant of IADN Interlaboratory Studies,

This letter is to invite your participation in the 1994 Interlaboratory Study for Trace Metals (Study 94-1), in support of the Integrated Atmospheric Deposition Network (IADN). This study will consist of spiked matrix samples, and will be in two parts. Part A will consist of spiked precipitation samples. Part B will consist of spiked air filters (used for sampling ambient air). Laboratories may choose to participate in only one part of the study if they do not routinely analyze both matrices.

Target metals for this study are Aluminum, Cadmium, Chromium, Copper, Lead, Zinc, Arsenic, and Selenium (both matrices).

Part A - Precipitation

Samples will be prepared unpreserved, and approximately 200 mL per sample will be provided to each participant in polyethylene (PET) bottles. There will be three samples in this study. If a special sample container is required by your laboratory, I am requesting that you ship a set of sample containers to me with your response form.

Target date for Part A: week of November 21, 1994.

Part B - Ambient Air Filters

For this part of the study, I am requesting that all participants provide me with four, clean (and pre-treated if appropriate) filters with their response form. This is necessary for this study, as the different IADN agencies (or other Air Toxics programs) use different filter types. Two of the filters will be spiked with the list of target elements, while the third filter will be a "travelling blank". One filter is a spare. Participants will be required to analyze the entire filter.

Target date for Part B: week of November 28, 1994.

Please return the attached response form to me at the above address, no later than November 10, 1994.

Please contact me at (416) 235-5842 if you require further explanations.

Sincerely,

Sylvia Cussion
Laboratory Quality Audit Scientist

INTERLABORATORY STUDY 94-1: TRACE METALS

Please return by November 10, 1994.

YES

NO

I will participate in Interlaboratory Study 94-1,
Part A, for the analysis of Trace Metals in Precipitation.

☐☐

I will participate in Interlaboratory Study 94-1,
Part B, for the analysis of Trace Metals on Air Filters.

☐☐

If you answered "No" for this study, you do not need to
answer the remainder of this questionnaire. However I
would appreciate receiving your "No" response.

Part A: Precipitation Samples

If a special container is required or the specified sample volume of 200 mL is insufficient, please
fill out the following and provide a set of sample containers with this response form.

a) Minimum sample volume required: _____

b) Sample container provided: _____

Part B: Ambient Air Samples

Type of filter provided: _____

Filter pretreatment: _____

Contact Person: _____

Phone and FAX: _____

Mailing Address:

Shipping Address:

125 Resources Rd.
Etobicoke, Ontario, M9P 3V6
Phone: (416) 235-5842
FAX: (416) 235-5744

November 21, 1994

Dear Participant of Interlaboratory Study 94-1, Part A,

Please find enclosed three precipitation samples for the analysis of Trace Metals. If you provided specific sample containers, the samples have been shipped to you in these containers. Please contact me immediately if there has been any damage during shipping, at (416) 235-5842.

The samples were prepared without a preservative. Those participants who provided sample containers that were pre-charged with a preservative will have received their samples preserved. For those participants receiving the samples unpreserved, please indicate on the accompanying report form any addition of preservative that is done upon sample reception.

Please report all results on the accompanying report form, including all questions regarding methodology, by December 23, 1994.

Thank you for your participation in this study. Please contact me if you have any questions or problems.

Your identification code is:

Sincerely,

Sylvia Cussion
Laboratory Quality Audit Scientist
(416) 235-5842
FAX (416) 235-5744

INTERLABORATORY STUDY 94-1, PART A
TRACE METALS FOR THE INTEGRATED ATMOSPHERIC DEPOSITION NETWORK

DUE DATE: DECEMBER 23, 1994

Identification Code: _____

Units: _____

Element	94-1-MET1		94-1-MET2		94-1-MET3	
	Dil. Factor	Result	Dil. Factor	Result	Dil. Factor	Result
Al						
As						
Cd						
Cr						
Cu						
Pb						
Se						
Zn						

Dil. Factor = Dilution Factor

SAMPLE PRESERVED: No _____ Yes _____

If Yes, Preservative Used: _____

SAMPLE TREATMENT PRIOR TO ANALYSIS: _____

INSTRUMENT USED FOR ANALYSIS: _____

125 Resources Rd
Etobicoke, Ontario, M9P 3V6
Phone: (416) 235-5842
FAX: (416) 235-5744

November 29, 1994

Dear Participant of Interlaboratory Study 94-1, Part B,

Please find enclosed three filter samples for the analysis of Trace Metals. Please contact me immediately if there has been any damage during shipping, at (416) 235-5842.

Please analyze the entire filter, as we cannot guarantee that the spiking solution spread evenly over the filter.

Please report all results on the accompanying report form, including all questions regarding methodology, by December 31, 1994.

Thank you for your participation in this study. Please contact me if you have any questions or problems.

Your identification code is:

Sincerely,

Sylvia Cussion
Laboratory Quality Audit Scientist
(416) 235-5842
FAX (416) 235-5744

INTERLABORATORY STUDY 94-1, PART B
TRACE METALS FOR THE INTEGRATED ATMOSPHERIC DEPOSITION NETWORK

DUE DATE: DECEMBER 31, 1994

Identification Code:

Units:

Element	94-1-MET4		94-1-MET5		94-1-MET6	
	Dil. Factor	Result	Dil. Factor	Result	Dil. Factor	Result
Al						
As						
Cd						
Cr						
Cu						
Pb						
Se						
Zn						

Dil. Factor = Dilution Factor

SAMPLE TREATMENT PRIOR TO ANALYSIS: _____

INSTRUMENT USED FOR ANALYSIS: _____

125 Resources Rd
Etobicoke, Ontario, M9P 3V6
Phone (416) 235-5700
FAX (416) 235-5744

April 10, 1995

To: Participants of Interlaboratory Study 94-1, Part A & B

Please find attached a copy of the Tables of results for Interlaboratory Study 94-1 for trace metals in support of the Integrated Atmospheric Deposition Network (IADN). I apologize for the delay in reporting these results, as there was a delay in receiving results from some of the participants.

Please review the results and let me know if there are any transcription errors by May 5, 1995. As you probably know, Ms. Sylvia Cussion is away on maternity leave and expects to be back by the middle of July, 1995. She will be working on the final report as soon as she returns.

Your Identification Code is:

Sincerely,

Sathi S. Selliah

